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National Food Safety Standard-Stabilized Chlorine Dioxide Solution

Report Categories:

FAIRS Subject Report

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Report Highlights:

On May 5, 2010, China notified the WTO of National Food Safety Standard: Food Additives – Stabilized Chloride Dioxide Solution as SPS/N/CHN/259. This measure applies to the definition and characterization of the food additive Stabilized Chloride Dioxide Solution. It specifies the scope, requirements and testing methods. The date for submission of final comments to China was May 20, 2010. The proposed date of entry was May 30, 2010. Contact information on where to send comments is inside the report. This report is an INFORMAL translation of this document.

Executive Summary:

On May 5, 2010, China notified the WTO of National Food Safety Standard: Food Additives – Stabilized Chloride Dioxide Solution as SPS/N/CHN/259. This measure applies to the definition and characterization of the food additive Stabilized Chloride Dioxide Solution. It specifies the scope, requirements and testing methods. The date for submission of final comments to China was May 20, 2010. The proposed date of entry was May 30, 2010.

Comments can be sent to the China WTO SPS Enquiry Point at: SPS@aqsiq.gov.cn.

This report contains an UNOFFICIAL translation of National Standard on Determination of Stabilized Chloride Dioxide Solution in Foods.

General Information:

BEGIN TRANSLATION

National food safety standard**Food additive Stabilized chlorine dioxide solution**

(Draft for soliciting opinions)

Promulgated on 2010-XX-XX
Executed from 201X-XX-XX

Foreword

Appendix A to the Standard is a normative appendix.

National food safety standard**Food additive Stabilized chlorine dioxide solution****1. Scope**

The Standard shall be applicable for stabilized ClO₂ solution made from food additive carbonate absorbing chlorine dioxide (ClO₂) gas. This production shall be activated by food additive HCl in use.

2. Cited normative documents

The documents cited in the Standard are imperative for application of the Standard. For the cited documents with date, only the version of the date shall be applicable for the Standard; for those without date, the latest version (including all revision lists) shall be applicable for the Standard.

3. Technical requirements

3.1 Requirements for sense evaluation: shall comply with the regulations of Table 1.
Table 1 Requirements for sense evaluation

Item	Requirements	Testing method
Color	No color or yellow	Take an amount of sample and put it in a beaker of 50ml, and observe the color and textural state in natural light.
Textural state	Transparent liquid without suspended matter	

3.2 Physical and chemical indicator: shall comply with the regulations of Table 2.

Table 2 Physical and chemical indicator

Item	Indicator	Testing method
Chlorine dioxide (ClO ₂), w/% ≥	2.0	A.4 of Appendix A
Density, ρ_{20} / (g/cm ³)	1.020~1.060	A.5 of Appendix A
pH value	8.2~9.2	A.6 of Appendix A
Arsenic (As), w/% ≤	0.0001	A.7 of Appendix A
Heavy metal (Calculated as per Pb), w/% ≤	0.0005	A.8 of Appendix A
Content of methanol, ρ / (g/100ml) ≤	0.04	A.9 of Appendix A

Appendix A Testing method (normative appendix)

A.1 Cautions

The reagent applied in this testing method is toxic and corrosive, so care shall be taken in operation! If any reagent spatters on skin, rinse it with water immediately, or go to hospital for treatment if serious; for combustible, no open flame shall be adopted for heating.

A.2 General conditions

Except otherwise specified, reagent and water applied in the Standard shall be pure reagent and water in Class III as specified in GB/T 6682-2008, and the required standard volumetric solution (VS), standard solution of impurity, and agent and product shall be prepared in accordance with HG/T 3696.1, HG/T 3696.2, and HG/T 3696.3.

A.3 Identification

A.3.1 Reagent

A.3.1.1 HCl solution: 1+2;

A.3.1.2 Calcium chloride solution: 100g/l.

A.3.2 Identification method

A.3.2.1 Sodium ion

Burn the platinum wire soaked in HCl on colorless flame till it becomes colorless; dip in a small amount of test solution and burn on colorless flame. Observe the flame through cobalt glass till it becomes bright yellow.

A.3.2.2 Carbonate

Add calcium chloride solution in the test solution and create white deposit, which can dissolve in HCL.

A.3.2.3 ClO₂

Add HCl solution in the test solution and create yellow gas, and simultaneously the test solution also becomes yellow.

A.4 Determination of content of ClO₂

A.4.1 Iodimetry (arbitration method)

A.4.1.1 Method abstract

Firstly, when pH ≤ 2, get rid of the disturbance of hypochlorite (ClO⁻) with malonic acid, and ClO₂ in the sample and all ClO₂⁻ reacts with KI; use standard solution of sodium thiosulfate to titrate the created iodine. Secondly, take the other sample, and adjust the solution till pH ≤ 2, and remove ClO₂ and chlorine with nitrogen gas; ClO₂⁻ in the sample reacts with KI. Use standard solution of sodium thiosulfate to titrate the created iodine. Calculate the difference between the 2 steps to obtain the actual content of ClO₂ in the sample.

A.4.1.2 Reagent

A.4.1.2.1 High-purity nitrogen gas;

A.4.1.2.2 HCl solution: 1+4;

A.4.1.2.3 KI solution: 100g/l;

A.4.1.2.4 Sulphuric acid solution: 1+8;

A.4.1.2.5 Malonic acid solution: 100g/l;

A.4.1.2.6 Standard VS of sodium thiosulfate: c (Na₂S₂O₃) ≈ 0.1 mol/l;

A.4.1.2.7 Starch indicating liquid: 5g/l.

A.4.1.3 Analysis steps

A.4.1.3.1 Preparation of sample solution A

Take and weigh about 10g of the sample, and keep the accuracy to 0.0002g; transfer to a measuring flask of 50ml, and dilute with water to the scale and shake up. This solution is marked as Test Solution A, used for determining the content of ClO₂.

A.4.1.3.2 Determination

Use a pipette to take out 10ml of test solution A and put into 2 iodine measuring flasks added with 50ml of water in advance, and add 2ml of HCl solution; cover and seal with water, and keep for 30min; and then conduct the determination as per the above first and second step. Firstly: add 3ml of malonic acid solution in an iodine measuring flask, and keep in shadow for 3min; add 10ml of sulphuric acid solution and 10ml of KI solution and mix up, and keep in shadow for 5min. Titrate with standard VS of sodium thiosulfate, add 1ml of starch indicating liquid near the end point, and titrate continuously till the blue color just disappears. Record the consumed volume as V₁.

Secondly: put the another iodine measuring flask in a fume cupboard with good ventilation condition; blow in high-purity nitrogen gas till the green yellow color disappears, and now keep blowing for 20 min; add 3ml of malonic acid solution and keep in shadow for 3min; add 10ml of sulphuric acid solution and 10ml of KI solution, and mix up and keep in shadow for 5min. Titrate with standard VS of sodium thiosulfate, and add 1ml of starch indicating liquid near the end point; titrate continuously till the blue color just disappears. Record the consumed volume as V₂.

A.4.1.4 Calculation

The content of ClO₂ shall be calculated as per the mass fraction of ClO₂ (w₁) and be expressed by % as shown in Formula A1:

$$w_1 = \frac{(V_1 - V_2) \times 1000 \times c}{m \times 10} \times 100 \quad \text{..... (A.1)}$$

In which:

V₁-- Consumed volume of standard VS of sodium thiosulfate by titration of the test solution in the first step (unit: ml)

V₂-- Consumed volume of standard VS of sodium thiosulfate by titration of the test solution in the first step (unit: ml)

c -- Accurate concentration of standard VS of sodium thiosulfate (unit: mol/l)

m-- Mass of the sample (unit: g)

M-- Molar mass of ClO₂ (1/5ClO₂) (unit: g/mol, M=13.49)

Take arithmetic mean of the parallel determination results as determination result, and the

absolute difference between 2 parallel determination results shall not be more than 0.2%.

A.4.2 Spectrophotometric method

A.4.2.1 Method abstract

At the point of 430nm, absorption of ClO_2 is shown. Use the spectrophotometric method for determination.

A.4.2.2 Reagent

A.4.2.2.1 Distilled water without chlorine

Add x sodium sulfite in the distilled water to determine the reagent with DPD residual chlorine, and there shall not be any color change for the reagent; distill now, and collect the distillate.

A.4.2.2.2 HCl solution: 1+4;

A.4.2.2.3 Standard stock solution of ClO_2

Preparation: add 300ml of water in Bottle A, and connect the glass tube at one end of Bottle A with air compressor, and another end with Bottle B. Bottle B is a borosilicate glass bottle with high strength, and there are 3 glass tubes at the neck. The 1st tube is inserted into the bottle to the point 5mm from the bottom, so as to introduce air; the 2nd tube is connected to a dropping funnel at the upper end, and lower end of the funnel extends below the liquid level; the lower end of 3rd tube leaves the liquid level, and the upper end is connected to Bottle C. dissolve 10g of sodium chlorite in 750ml of water and pour into Bottle B. In the separating funnel, there are 20ml of sulphuric acid solution (1+10); in Bottle C, saturated solution of sodium chlorite. Bottle D is a collection bottle of borosilicate glass of 2l, where there are 1500ml of water to absorb the created ClO_2 , and the residual air shall be exhausted from the pipe. The whole set of device shall be put into a fume cupboard (See Figure A.1).

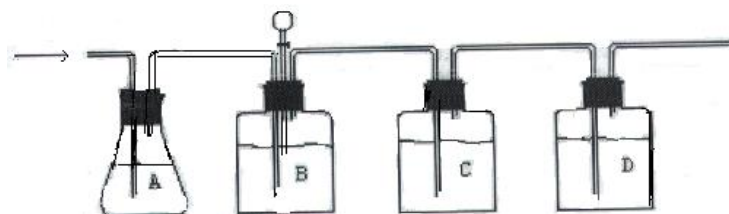


Figure A.1 Creation and absorption device of ClO_2

Start up air compressor, and enable the air to pass through the whole set of device evenly. Add 5ml of sulphuric acid solution ever 5 minutes with a separating funnel. After add for the last time, keep the air flow for 30 min. put the obtained yellow standard solution of ClO_2 in a brown bottle and keep it below 4°C . The concentration shall be 250 mg/l~600 mg/l.

Determine the concentration of ClO_2 in the stock solution by the method specified in A.4.1 (determine the concentration in the stock solution before use every time).

A.4.2.2.4 Standard solution of ClO_2 : in 1ml of the solution, the content of ClO_2 shall be 0.25 mg.

According to the concentration of ClO_2 in the stock solution, absorb an amount of standard stock solution of ClO_2 , and dilute with distilled water without chlorine. This solution shall be prepared just before use.

A.4.2.3 Analysis steps

A.4.2.3.1 Drawing of working curve

Take out 0.0ml, 10.0ml, 20.0ml, 40.0ml, 60.0ml, 80.0ml, and 100.0ml of standard solution of ClO_2 and put into 7 measuring flask of 100ml respectively, and dilute with distilled water without chlorine to the scale and shake up. In a wavelength of 430 nm, select a colorimetric tube of 1cm, adjust to zero with distilled water without chlorine, and measure the absorbance. Deduct the absorbance of the blank test solution from that of the standard solution; take the mass of ClO_2 (mg) as the abscissa and the relevant absorbance as the ordinate to draw the working curve.

A.4.2.3.2 Determination

Take and weigh 0.7g of the sample and keep the accuracy to 0.0002g; put in 10ml of HCl solution, and transfer to a measuring flask of 100ml; dilute with distilled water without chlorine to the scale and shake up. In a wavelength of 430nm, select a colorimetric tube of 1cm and adjust to zero with distilled water without chlorine to measure the absorbance. Find out the mass of ClO₂ in the test solution from the working curve.

A.4.1.4 Calculation

The content of ClO₂ shall be calculated as per the mass fraction of ClO₂ (w₁) and be expressed by % as shown in Formula A2:

$$w_1 = \frac{m_1 \times 10^{-3}}{m} \times 100 \dots\dots\dots (A.2)$$

In which:

m₁-- Mass of ClO₂ in the test solution found out from the working curve (unit: mg)

m -- Mass of the sample (unit: g)

Take arithmetic mean of the parallel determination results as the reported result, and the absolute difference among the parallel determination results shall not be more than 0.02%.

A.5 Determination of density

A.5.1 Instrument and equipment

As specified in 2.3.3.2 of GB/T 4472-1984.

A.5.2 Analysis steps

As specified in 2.3.3.3 of GB/T 4472-1984.

Take arithmetic mean of the parallel determination results as the reported result, and the absolute difference among the parallel determination results shall not be more than 0.003g/cm³.

A.6 Determination of pH value

A.6.1 Instrument and equipment

As specified in Chapter 5 of GB/T 9724-2007.

A.6.2 Analysis steps

Pour the sample into a beaker, and determine in accordance with Chapter 6 of GB/T 9724-2007.

Take arithmetic mean of the parallel determination results as the reported result, and the absolute difference among the parallel determination results shall not be more than 0.2 ph unit.

A.7 Determination of arsenic

Take and weigh 1.00g±0.01g of the sample and put into a beaker of 100ml; add 8ml of HCl solution (1+3). Boil it on an electric furnace till near dry, and add a small amount of water to be test solution.

Use a pipette to take out 1.0ml of standard solution of arsenic as the standard (in 1ml of the solution, the content of arsenic shall be 1μg], and then determine in accordance with GB/T 5009.76-2003.

A.8 Determination of content of heavy metal

Take and weigh 1.00g±0.01g of the sample and put into a beaker of 100m, and add 8ml of HCl solution (1+3); put on an electric furnace and boil till near dry, and then take down and cool to room temperature; add a small amount of water and add a drop of phenolphthalein indicating liquid, adjust with ammonia solution and HCl solution till that the pH value reaches to neutral (i.e. red color of phenolphthalein just disappears) to be as test solution.

Use a pipette to take out 0.5ml of standard solution of Pb [in 1ml of the solution, the content of Pb shall be 10μg] to be as the standard, and determine in accordance with Chapter 6 in GB/T 5009.74-2003.

A.9 Determination of content of methanol

A.9.1 Reagent

A.9.1.1 Potassium permanganate- phosphoric acid solution:

Take and weigh 3g of potassium permanganate and add in mixed liquid of 15ml of phosphoric acid and 70ml of water; after dissolving, add water to 100ml. Store it in a brown bottle.

A.9.1.2 Oxalic acid-sulphuric acid solution:

Take and weigh 5g of oxalic acid without water or 7g of oxalic acid with 2 crystal water, and dissolve and dilute with sulphuric acid solution (1+1) to 100ml.

A.9.1.3 Fuchsin-sulfurous acid solution:

Take and weigh 0.1g of alkaline fuchsin; after porphyrization, add 60ml of water of 80°C totally for 3 times, and grind simultaneously to dissolve it; use a burette to suck the solution in the top layer and filter into a measuring flask of 100ml. after cooling, add 10ml of sodium sulfite solution (100g/l) and 1ml of HCL, and add again water to the scale and mix up adequately; keep for a night. If the solution is with a color, add a small amount of active carbon and mix up, and then filter and store in a brown bottle; keep in shadow. If the solution is with red color, prepare newly.

A.9.1.4 Standard stock solution of methanol: in 1ml of the solution, the content of methanol (CH₃OH) shall be 10 mg.

Take and weigh 1.000g of CH₃OH and put into a measuring flask of 100ml, and add water and dilute to the scale and shake up.

A.9.1.5 Standard solution of CH₃OH: in 1ml of the solution, the content of CH₃OH shall be 1 mg.

Use a pipette to take out 10ml of standard stock solution of CH₃OH (A.9.1.4) and put into a measuring flask of 100ml; add water to dilute to the scale and shake up. This solution shall be prepared just before use.

A.9.2 Analysis steps

Use a pipette to take out 1.00ml of the sample and put into a colorimetric tube of 25ml with cover, and add water to 15ml to be as test solution.

For the standard, use a pipette to take out 0.40ml of standard solution of CH₃OH (A.9.1.5) and put into another colorimetric tube, and add water to 15ml.

Add 2ml of potassium permanganate- phosphoric acid solution into the two colorimetric tubes respectively, mix up, and keep for 10min; add 2ml of oxalic acid-sulphuric acid solution, and mix up till the color is faded; and then add 5ml fuchsin- sulfurous acid solution, and dilute with water to the scale and shake up. Keep for 30min in a condition of more than 20°C, ensuring that that the sample color shall not be deeper than the standard.

END TRANSLATION